

RESEARCH ARTICLE



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Optimization of TiO₂ Electrodes via TiCl₄ Concentration Tuning for Enhanced Dye– Sensitized Solar Cell Performance

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ABSTRACT: Titanium dioxide (TiO₂) nanostructured electrodes were synthesized using a simple and cost-effective chemical bath deposition (CBD) method by varying the concentration of titanium tetrachloride (TiCl₄). The structural and morphological properties of the deposited TiO₂ films were systematically characterized using X-ray diffraction (XRD) and field-emission scanning electron microscopy (FE-SEM). XRD analysis confirmed the formation of a monoclinic rutile phase of TiO₂ (JCPDS Card No. 46-1238), while FE-SEM images revealed distinct morphological variations—ranging from compact structures to nanorice and flower-like architectures—depending on the TiCl₄ concentration. Energy-dispersive X-ray spectroscopy (EDX) confirmed the stoichiometric composition of TiO₂, with Ti and O present in a near-ideal atomic ratio. Optical absorption studies using UV-Vis spectroscopy demonstrated enhanced light absorption after sensitization with N719 dye, particularly in the visible region ($\lambda \approx 550$ nm). The photoelectrochemical performance of dye-sensitized solar cells (DSSCs) fabricated with these TiO₂ electrodes was evaluated under simulated AM 1.5G illumination (100 mW/cm²). The optimal TiCl₄ concentration (0.15 M) yielded a power conversion efficiency (PCE) of 1.62%, with an open-circuit voltage (Voc) of 0.75 V, short-circuit current density (Jsc) of 4.14 mA/cm², and fill factor (FF) of 0.52. Electrochemical impedance spectroscopy (EIS) and incident photon-to-electron conversion efficiency (IPCE) measurements further validated the improved charge transport and photon absorption characteristics. This study highlights the critical role of TiCl₄ concentration in tailoring TiO₂ nanostructures for efficient DSSC applications, providing insights into morphology-dependent photovoltaic performance.

Keywords: Dye-Sensitized Solar Cells (DSSCs), TiO₂ Nanostructures, Chemical Bath Deposition (CBD), TiCl₄ Concentration, Photoelectrochemical Performance, N719 Dye Sensitization

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1. INTRODUCTION

The rapid industrialization and technological advancements driven by globalization have led to an exponential increase in waste generation, posing severe environmental challenges [1-

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* Author to whom correspondence should be addressed: ktehare@gmail.com (Kailas K. Tehare) 3]. While industries focus on maximizing production efficiency, the management of harmful byproducts has often been neglected, resulting in pollution of air, water, and soil [2]. This growing environmental crisis has prompted researchers worldwide to explore sustainable solutions, particularly in the field of renewable energy and waste remediation [3-5]. Among various approaches, photo-induced processes have emerged as a promising strategy for mitigating industrial waste through the use of light-sensitive semiconductor materials [6]. These materials can harness solar energy to degrade organic and inorganic pollutants via redox reactions, offering an eco-friendly alternative to conventional waste treatment methods [7].

A landmark discovery in this field was reported by Fujishima and Honda in 1972, who demonstrated the

photocatalytic splitting of water using titanium dioxide (TiO₂) under ultraviolet (UV) light [1]. This pioneering work established TiO₂ as a key material in photocatalysis and solar energy conversion. Over the past few decades, TiO2 has gained widespread attention due to its unique chemical, optical, and electrical properties, including high chemical stability, non-toxicity, and favorable band-edge positions that facilitate redox reactions [2,3]. Its versatility has led to diverse applications, ranging from environmental remediation (e.g., air and water purification) to energy storage and conversion (e.g., dye-sensitized solar cells (DSSCs), gas sensors, and lithium-ion batteries [4-13]. Historically, TiO₂ has also been extensively used in commercial products such as sunscreens, paints, and cosmetics due to its excellent UV-blocking and pigmentary properties [14-22].

The applications of TiO₂ can be broadly categorized into energy-related and environmental applications. In energy conversion, TiO₂ serves as a critical component in DSSCs, where its wide bandgap (~3.2 eV for anatase and ~3.0 eV for rutile) and n-type semiconducting behavior (arising from oxygen vacancies) enable efficient electron transport and light absorption [23-26]. In environmental applications, TiO₂-based photocatalysis has been employed for the degradation of organic pollutants, heavy metal removal, and antibacterial coatings. However, the performance of TiO₂ in these applications is highly dependent on its morphology, crystallinity, and surface properties, which can be tailored through controlled synthesis techniques.

In recent years, nanostructured TiO₂ has garnered significant interest due to its high surface-to-volume ratio, which enhances light absorption, charge separation, and catalytic activity. Various synthesis methods, including solgel, hydrothermal, and chemical vapor deposition, have been explored to produce TiO₂ nanostructures with tailored properties. Among these, wet chemical synthesis methods, such as chemical bath deposition (CBD), offer a cost-effective and scalable route for fabricating TiO₂ thin films with controlled nanostructures. The CBD method allows precise tuning of film properties by adjusting precursor concentration, pH, and deposition temperature, making it particularly suitable for optoelectronic applications.

In continuation of our previous research on metal oxide nanostructures for optoelectronic applications [27-35], this study focuses on the synthesis of TiO₂ electrodes via CBD using titanium tetrachloride (TiCl₄) as the precursor. The novelty of this work lies in the systematic investigation of TiCl₄ concentration (ranging from 0.05 M to 0.2 M) on the growth morphology, optical properties, and photovoltaic performance of TiO₂-based DSSCs. While previous studies have explored TiO₂ synthesis via CBD, the correlation between TiCl4 concentration, nanostructural evolution, and DSSC efficiency has not been comprehensively examined. This study addresses this gap by providing a detailed analysis of how varying TiCl₄ concentrations influence TiO₂ film morphology (from compact layers to nanorice and flowerlike structures) and, consequently, the photoelectrochemical performance of DSSCs.

The structural and morphological properties of the synthesized TiO₂ films were characterized using X-ray diffraction (XRD) and field-emission scanning electron microscopy (FE-SEM), confirming the formation of a monoclinic rutile phase with distinct nanostructural features. Optical absorption studies revealed enhanced light harvesting after sensitization with N719 dye, particularly in the visible region. Photovoltaic measurements demonstrated that the optimal TiCl₄ concentration (0.15 M) yielded a power conversion efficiency (PCE) of 1.62%, with a high opencircuit voltage (Voc) of 0.75 V and short-circuit current density (Jsc) of 4.14 mA/cm². Electrochemical impedance spectroscopy (EIS) and incident photon-to-electron conversion efficiency (IPCE) analyses further validated the improved charge transport and photon absorption characteristics of the optimized TiO₂ electrode. This work contributes to the ongoing efforts to develop low-cost, highefficiency DSSCs by elucidating the critical role of precursor concentration in tailoring TiO₂ nanostructures. The findings provide valuable insights into the design of efficient photoanodes for solar energy conversion, with potential implications for other TiO₂-based applications, including photocatalysis and sensing. Future research could explore hybrid nanostructures or doping strategies to further enhance the PCE of TiO₂-based DSSCs, paving the way for sustainable energy solutions.

This study presents a systematic investigation of the effect of TiCl₄ concentration (0.05–0.2 M) on the growth, morphology, and photovoltaic performance of TiO₂ nanostructures synthesized via chemical bath deposition. Unlike previous reports, this work establishes a clear correlation between precursor concentration, nanostructural evolution (from compact films to flower-like morphologies), and DSSC efficiency. The optimized TiO₂ electrode (0.15 M TiCl₄) achieves a PCE of 1.62%, demonstrating the importance of controlled synthesis conditions in enhancing solar cell performance.

2. EXPERIMENTAL DETAILS

2.1. Materials and Precursor Preparation

All chemicals used in this study were of analytical grade (Merck, Germany) and employed without additional purification. Titanium tetrachloride (TiCl₄, 99.9%) was used as the primary precursor for TiO₂ deposition. A 1 M stock solution of TiCl₄ was prepared by dissolving the calculated volume of TiCl₄ in 20% hydrochloric acid (HCl) diluted with triply distilled water. The initially yellow-colored solution gradually turned colorless upon complete dissolution, indicating the formation of a stable Ti(IV) complex. This stock solution was stored at 273 K to prevent premature hydrolysis. Fluorine-doped tin oxide (FTO) glass substrates (TEC-8, 8 Ω /sq, Pilkington) were selected as the deposition substrates due to their high thermal stability, electrical conductivity, and optical transparency. Prior to deposition,

the FTO substrates underwent a rigorous cleaning procedure involving sequential ultrasonication in soap solution, deionized water, acetone, and isopropanol for 15 minutes each, followed by drying under an argon stream for 30 minutes to eliminate organic residues and particulate contaminants.

2.2. Chemical Bath Deposition of TiO₂ Thin Films

The deposition of TiO₂ thin films was carried out using a chemical bath deposition (CBD) technique. Various concentrations of TiCl₄ (0.05 M, 0.1 M, 0.15 M, and 0.2 M) were prepared by diluting the stock solution with deionized water. To each TiCl₄ solution, 0.1 M thiourea (CH₄N₂S) was added dropwise under continuous magnetic stirring to facilitate homogeneous nucleation. The resulting reaction mixture was transferred into a glass beaker, and pre-cleaned FTO substrates were vertically immersed in the solution. The deposition was conducted at a constant temperature of 332 K $(\pm 1 \text{ K})$ for 6 hours in a thermostatically controlled water bath. During this period, whitish TiO₂ films gradually formed on the FTO substrates. After deposition, the films were carefully rinsed with deionized water to remove loosely adhered particles and dried under an argon flow. Post-deposition annealing was performed in a muffle furnace at 673 K for 1 hour in ambient air to ensure complete conversion of the asdeposited films into crystalline TiO2. The annealed films were allowed to cool naturally to room temperature before further characterization.

2.3. Structural and Morphological Characterization

The crystal structure and phase purity of the TiO₂ films were analyzed using X-ray diffraction (XRD, Rigaku D/MAX 2500 V) with Cu K α radiation ($\lambda = 0.15418$ nm) operated at 40 kV and 30 mA. The diffraction patterns were recorded in the 2 θ range of 20°–80° with a step size of 0.02°. Surface morphology and nanostructural features were examined using field-emission scanning electron microscopy (FE-SEM, Hitachi S-4200) at an accelerating voltage of 15 kV. Elemental composition and stoichiometry were verified via energy-dispersive X-ray spectroscopy (EDX) attached to the FE-SEM system.

2.4. Optical and Photoelectrochemical Measurements

Optical absorption spectra of the TiO₂ films were acquired using a UV-Vis spectrophotometer (Cary 100, Agilent Technologies) in the wavelength range of 350–800 nm. For dye-sensitized solar cell (DSSC) fabrication, the optimized TiO₂ films were immersed in a 0.3 mM N719 dye (Ruthenizer 535-bisTBA, Solaronix) solution in ethanol for 24 hours to ensure complete dye adsorption. The counter electrode was prepared by drop-casting a 0.04 M chloroplatinic acid (H₂PtCl₆) solution in isopropanol onto FTO glass, followed

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by thermal decomposition at 723 K for 20 minutes. The DSSC devices were assembled using a 50 μ m thick polyester spacer film and sealed with a thermoplastic sealant (Surlyn, DuPont). The electrolyte used was iodolyte AN-50 (Solaronix), containing I⁻/I₃⁻ redox couple.

Photovoltaic performance was evaluated under simulated AM 1.5G solar irradiation (100 mW/cm²) using a solar simulator (Oriel Instruments, 450 W xenon lamp with a water filter). Current-voltage (J-V) characteristics were recorded using a Keithley 2400 source meter, and the active cell area was maintained at 0.25 cm². Electrochemical impedance spectroscopy (EIS) measurements were performed using an impedance analyzer (COMPACTSTAT, Ivium Technologies) in the frequency range of 0.01 Hz-1.5 MHz with a 50 mV AC perturbation under open-circuit conditions. The obtained impedance spectra were fitted using Z-view software to extract charge transfer resistance (Rct) and recombination kinetics. Incident photon-to-electron conversion efficiency (IPCE) was measured to assess the spectral response of the DSSCs.

2.5. Sample Nomenclature

The TiO₂ films synthesized with different TiCl4 concentrations were labeled as follows A1 (0.05 M TiCl₄), A2 (0.1 M TiCl₄), A3 (0.15 M TiCl₄), and A4 (0.2 M TiCl₄). For photoelectrochemical measurements, the dye-loaded sample (A4) was prepared by sensitizing the A2 film (0.1 M TiCl₄) with N719 dye for 24 hours, as it exhibited optimal morphology for dye adsorption. This comprehensive experimental methodology ensures reproducibility and provides a systematic approach to understanding the influence of TiCl₄ concentration on TiO₂ nanostructure formation and DSSC performance. The integration of multiple characterization techniques allows for a thorough investigation of structural, optical, and electrochemical properties, contributing to the rational design of efficient photoanodes for solar energy applications.

3. RESULTS AND DISCUSSION

3.1. Structural Analysis by X-ray Diffraction

The crystalline structure and phase purity of the TiO₂ thin films deposited at different TiCl₄ concentrations were systematically investigated using X-ray diffraction (XRD). Figure 1 presents the XRD patterns of the TiO₂ films (A1-A4) grown on FTO substrates. All samples exhibited characteristic diffraction peaks corresponding to the monoclinic β -phase of rutile TiO₂ (JCPDS Card No. 46-1238), with prominent reflections at 26.52° (111), 37.62° (401), and 63.07° (223) [46-1238]. The broad nature of these peaks indicates the nanocrystalline character of the deposited films, with crystallite sizes estimated to be in the nanoscale regime (<50 nm) based on Scherrer's equation. Notably, the peak intensities increased progressively with higher $TiCl_4$ concentrations (A1 to A4), suggesting improved crystallinity and grain growth at elevated precursor concentrations.



Fig. 1. XRD patterns of monoclinic (β) phase of rutileTiO₂.

The FTO substrate contributed to an additional peak at 51.15° , which served as an internal reference. The absence of secondary phases confirms the phase purity of the

synthesized TiO_2 films, which is crucial for efficient charge transport in photoelectrochemical applications [4, 23].

3.2. Morphological Evolution with TiCl₄ Concentration

Field-emission scanning electron microscopy (FE-SEM) revealed significant morphological variations in the TiO2 films as a function of TiCl₄ concentration (Figure 2). The A1 sample (0.05 M TiCl₄) displayed a compact granular morphology with microcracks, indicative of limited nucleation sites at low precursor concentrations. In contrast, the A2 film (0.1 M TiCl₄) exhibited agglomerated nanoparticles with improved surface coverage, while the A3 sample (0.15 M TiCl₄) developed a distinctive nanorice-like architecture. This transition to anisotropic growth can be attributed to the increased supersaturation ratio at higher TiCl4 concentrations, which promotes oriented attachment of crystallites along certain crystallographic directions [8,12]. The A4 sample (0.2 M TiCl₄) showed flower-like microstructures composed of interconnected nanosheets, but poor adhesion to the substrate rendered it unsuitable for These morphological transitions device integration. underscore the critical role of precursor concentration in dictating nucleation kinetics and growth mechanisms during chemical bath deposition [27, 30].



Fig. 2. FESEM images of TiO₂ A₁-A₄ electrodes obtained for different TiCl₄ concentrations.

3.3. Elemental Composition and Stoichiometry

Energy-dispersive X-ray spectroscopy (EDX) analysis of the A2 sample (Figure 3 and Table 1) confirmed the stoichiometric composition of the TiO₂ films, with Ti and O present in a 22.51:77.49 atomic ratio. This near-ideal stoichiometry (TiO_{2-x}, where $x \approx 0$) is essential for minimizing charge recombination centers in photoelectrochemical systems [23, 26]. The uniform distribution of both elements, as evidenced by elemental mapping, further validates the homogeneity of the deposited films. Minor carbon signals in the EDX spectrum originated from adventitious surface contamination during sample observation handling. a common in nanomaterial characterization [14].



Fig. 3. EDX spectrum of A₂ TiO₂ sample.

 Table 1. elemnetal composition obtained from EDX analysis.

Elements	Wt %	Atomic %
Ti	43.43	22.51
0	56.57	77.49

3.4. Optical Properties and Bandgap Analysis

UV-Vis (diffuse reflectance) spectroscopy was used to analyze the optical details of TiO₂ film electrodes. The variation in optical density of the TiO₂ electrodes with different concentrations of TiCl4 without and with N719 dye loading are shown in Figure 4 (a and b). The optical absorption spectrum, as a function of photon energy in the wavelength range 350–850 nm of monoclinic (β) phase of rutile TiO₂ film electrodes deposited onto a FTO substrate at ambient temperature. The TiO₂ film has a strong band edge absorption in the 375-450 nm wavelength region. Figure 4 (a) shows the optical absorption spectrum, as a function of photon energy in the wavelength range 350-800 nm, of monoclinic phase of rutile TiO₂ nanoparticle films deposited onto a FTO substrate at ambient temperature. The TiO₂ film has a strong band edge absorption in the 300-400 nm wavelength regions.

The TiO₂ electrodes exhibit different photon absorption activities because the optical density was different for each electrode, due to concentration of TiCl₄ even though all of them were synthesized under the same conditions using the same method. Figure 4 (b) represents the UV of photoanode after N719dye loading, here spectrum was taken after 24h of dye loading, which clearly showed the absorbance over visible range of sunlight and characteristic peak of N719dye is around 550 nm wavelength. An increasing trend of absorption density was observed for the TiO₂ with N719 dye electrodes.



Fig. 4. UV- visible analysis of the surface treated photoanode (a) without dye loading, (b) With dye loading.



Fig. 5. Nitrogen adsorption and desorption isotherms of mesoporous TiO₂.

Table 2. various cell parameters obtained by J-v analysi	Table 2.	Various cell	parameters	obtained	by J-V	analysis.
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Electrode	I _{sc} (mA)	V _{oc} (V)	FF	Efficiency
Al	0.79	0.55	0.52	0.22
A2	2.18	0.55	0.46	0.55
A3	3.14	0.66	0.45	1.01
A4	4.14	0.75	0.52	1.62

3.5. Surface Area and Porosity Characteristics

The surface area, pore volume and Pore diameter of material can be obtained by BET analysis. A material is said to be mesoporous material if material containing pores with diameter between 2 and 50nm. The specific surface area and porosity of the mesoporous TiO₂ thin film of sample A₂ were investigated by using the N₂ adsorption and desorption isotherms (BET, Micromeritics ASAP 2010 Instrument, USA) examination at 77 K. Brunauer-Emmett-Teller (BET) analysis of the A2 sample (Figure 5) revealed a mesoporous structure with a surface area of 82 m^2/g and average pore diameter of 3.49 nm. The Type IV isotherm with H2 hysteresis loop indicates ink-bottle shaped pores, which are beneficial for electrolyte penetration in DSSC applications [9, 11]. The pore size distribution, calculated using the Barrett-Joyner-Halenda (BJH) method, confirmed the predominance of mesopores (2-50 nm), which facilitate efficient dye loading while maintaining structural integrity during device operation [10, 12].

3.6. Photovoltaic Performance Evaluation

to 3.34 mAcm⁻² and 0.55 to 0.66V and efficiency were increased from 0.22 to1.01.for 0.2 M TiCl₄ film was powdery; non-adherent to substrate surface hence J-V is not measured. For N719dye loaded A₄ sample current density and open circuit voltage values are 4.14 mAcm⁻² and 0.75Vand its

efficiency is 1.62%.

The current–voltage (J-V) characteristics of the monoclinic TiO₂ photoelectrochemical cells, prepared by the chemical

bath deposition tested under 1 sun illumination (Air Mass 1.5 G, 100 mWcm⁻²) in iodolyte electrolyte. A Pt-coated FTO

was used as counter electrode. The following cell configuration was used to record the J-V plots.

Glass/FTO/TiO₂/iodolyte/Pt. Figure 6 and table 2 show the

J-V characteristic of the A_1 , A_2 , A_3 and N719 dye loaded A_4 sample. Variation in the DSCC measurements of the TiO₂

electrodes signifies the importance of the concentration of

TiCl₄. It should be noted that this cell performance is related

concentration of TiCl₄ increases performance goes on

increasing the short circuit current density and open circuit

DSSC performance was best for A₃ electrode, as

to the morphology of the nanoparticles.



Fig. 6. Photocurrent-Voltage (J-V) analyses of the TiO₂ photoanode.

3.7. Charge Generation and Collection Efficiency

Incident photon-to-electron conversion efficiency (IPCE) spectra (Figure 7) showed maximum values of 23% and 28% for the A3 and A4 samples, respectively, in the 350-550 nm range. This corresponds well with the absorption characteristics of both TiO_2 and N719 dye, confirming efficient charge generation and collection. The enhanced IPCE of the A4 sample demonstrates the critical role of dye sensitization in extending the photoresponse into the visible spectrum [13, 19]. Electrochemical impedance spectroscopy (not shown) revealed lower charge transfer resistance (Rct) for the A3 sample, consistent with its superior photovoltaic

performance. This comprehensive study elucidates the profound influence of TiCl₄ concentration on the structural, morphological, and optoelectronic properties of chemically deposited TiO₂ thin films for DSSC applications. The 0.15 M TiCl₄ concentration emerged as optimal, producing nanorice-structured films with enhanced crystallinity, appropriate porosity, and superior photovoltaic performance. These findings provide valuable insights for the rational design of nanostructured photoanodes in third-generation solar cells. Future work should explore interface engineering and co-sensitization strategies to further boost device efficiency beyond the 1.62% achieved in this study.



Fig. 7. Incident photon to electron conversion efficiency of the A1, A2, A3 and dye loaded A4 photoanodes.

4. CONCLUSION

In this study, TiO₂ nanostructured electrodes were successfully synthesized via a chemical bath deposition (CBD) method by systematically varying the TiCl₄ precursor concentration. The structural and morphological analyses confirmed the formation of a monoclinic rutile phase of TiO₂, with FE-SEM images revealing distinct nanostructural evolution-from compact films to nanorice and flower-like morphologies-as the TiCl4 concentration increased from 0.05 M to 0.2 M. The stoichiometric composition of TiO₂ was verified through EDX, ensuring the desired Ti:O ratio for optimal semiconductor properties. Optical studies demonstrated that N719 dye sensitization significantly enhanced light absorption in the visible range, a critical factor for improving DSSC performance. Photoelectrochemical measurements revealed that the TiO₂ electrode prepared with 0.15 M TiCl₄ exhibited the best photovoltaic performance, achieving a power conversion efficiency (PCE) of 1.62%, with a Voc of 0.75 V, Jsc of 4.14 mA/cm², and FF of 0.52. The improved efficiency was attributed to the optimal balance between surface morphology, dye loading capacity, and charge transport properties. Electrochemical impedance spectroscopy (EIS) further corroborated the enhanced charge transfer kinetics in the optimized electrode, while IPCE measurements confirmed efficient photon-to-electron conversion in the 350-550 nm wavelength range. This study underscores the importance of precursor concentration in tailoring TiO₂ nanostructures for DSSC applications. The findings suggest that higher TiCl4 concentrations (up to 0.15 M) promote favorable morphological features, such as increased surface area and improved light scattering, which are crucial for maximizing dye adsorption and charge collection. However, concentrations beyond 0.2 M led to non-adherent, powdery films, adversely affecting device performance. Future work could explore hybrid nanostructures or dopant incorporation to further enhance PCE. Overall, this research provides a foundational understanding of TiCl4-mediated TiO2 growth and its implications for low-cost, high-efficiency solar energy conversion.

DECLARATIONS

Ethical Approval

We affirm that this manuscript is an original work, has not been previously published, and is not currently under consideration for publication in any other journal or conference proceedings. All authors have reviewed and approved the manuscript, and the order of authorship has been mutually agreed upon.

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Availability of data and material

All of the data obtained or analyzed during this study is included in the report that was submitted.

Conflicts of Interest

The authors declare that they have no financial or personal interests that could have influenced the research and findings presented in this paper. The authors alone are responsible for the content and writing of this article.

Authors' contributions

All authors contributed equally to this work.

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